

# A CARBON ARC APPARATUS FOR PRODUCTION OF NANOTUBES IN MICROGRAVITY

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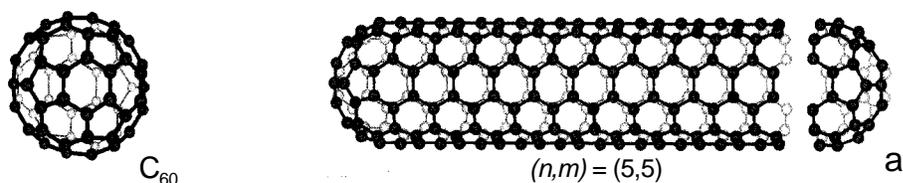
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## ABSTRACT

Although many methods are available for production of single-walled carbon nanotubes (SWNTs), the conventional carbon arc process remains the most popular due to its simplicity and large production rate. However, high temperatures inside the carbon arc generate strong buoyancy driven convection, and it is hypothesized that the non-uniform environment created by this flow will have large effects on the growth and morphology of SWNTs produced by the arc process. Indeed, using normal gravity experiments, Marin *et al.*<sup>1</sup> have demonstrated that changes in the buoyant convection plume produced by altering the arc electrode orientation can be used to change the diameter distribution of the SWNTs produced; an effect they attribute to changes in the temperature of the local nanotube growth environment. While these experiments present convincing evidence that buoyant convection has a strong effect on nanotube growth, normal gravity experiments are severely limited in scope. The ideal way to study the effect of buoyancy on SWNT production is to remove it completely. Toward this goal, a microgravity carbon arc reactor has been designed for use in the NASA Glenn 2.2 and 5 second drop towers. Although simple in principle, conventional carbon arc machines, which generally employ large reaction chambers and require heavy duty welding power supplies capable of supplying kilowatts of power, are not suitable for microgravity experiments. Here we describe a miniature carbon arc machine for SWNT production that fits into a conventional drop rig for use on the NASA Glenn 2.2 and 5 second drop towers, but that has a performance (production rate) that is better than most large ground-based machines.

## INTRODUCTION

There is currently a large interest in the synthesis and characterization of carbon nanotubes for many useful applications. Carbon nanotubes were first discovered in 1991 by Iijima<sup>2</sup> who observed the presence of nanometer sized multi-walled carbon nanotubes (MWNTs) on the graphite electrodes used for fullerene production. Addition of a transition metal catalyst was subsequently shown to produce single-walled nanotubes<sup>3</sup> (SWNTs) such as the C<sub>60</sub> sized nanotube illustrated in Figure 1. Unlike conventional carbon fibers, the SWNT structure is atomically perfect, thus producing defect free fibers that have many unique electronic and physical properties<sup>4</sup>. For example, with a predicted Young's modulus of ~1 Tera-Pascal, SWNTs represent the strongest known type of carbon fiber and form the ideal basis for new composite materials. Calculations show that a carbon nanotube-based cable could have one hundred times the strength of steel while having just one-sixth of the weight. Carbon fiber composites made from nanotubes would save significant weight in spacecraft and aircraft structures. In addition to their mechanical properties, nanotubes also have interesting electronic properties, which are



**Figure 1.** Comparison of  $C_{60}$  fullerene and its single-walled nanotube analogue.

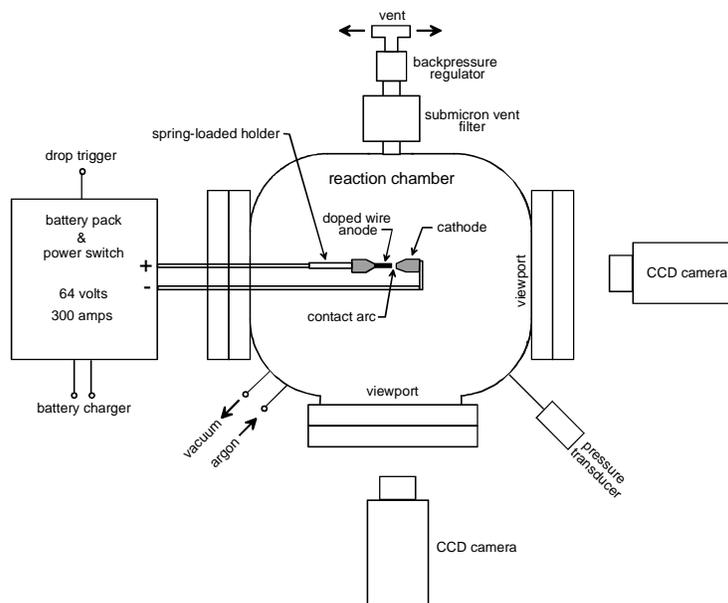
dependent upon the tubes morphology. Some tubes have conducting electronic structures and can be envisioned as molecular “quantum” wires, while others are semiconducting and can be used to fabricate the world’s smallest “single molecule” transistors. Other promising uses for SWNTs include tips for atomic force microscopy (AFM) and possibly hydrogen storage.

There are currently several methods for the production of single-walled carbon nanotubes, including: the carbon arc<sup>5</sup>, laser vaporization of graphite<sup>6</sup>, chemical vapor decomposition<sup>7</sup> (CVD), high-pressure disproportionation of  $CO$ <sup>8</sup>, and flame synthesis<sup>9</sup>. All rely on catalytic growth of the nanotube from either carbon or hydrocarbon vapor in the presence  $Co/Ni$  or other transition metal catalysts. As vaporized carbon (or hydrocarbon) and metal catalyst atoms cool, they condense into small nanometer sized clusters that continually collide and grow. When the metal carbide clusters produced from the transition metal catalyst become super-saturated with carbon, the carbon re-crystallizes as nanotubes. The metal particle remains on the head of the growing nanotube and channels the remaining carbon it encounters into it. Studies performed on the vapor plume produced during laser ablation of graphite suggest that nanotube growth can continue as long as the catalyst particle remains saturated with carbon and the temperature remains high enough for carbon to efficiently diffuse through the particle<sup>10</sup>. The formation of a nanotube is undoubtedly a strong function of the time/temperature/concentration history of the growing tube and its precursors, and buoyancy produces an uncontrolled environment that makes estimation and optimization of these critical factors difficult. Microgravity conditions provide a much more controlled environment for measurement, modeling, and optimization of these parameters.

## EXPERIMENTAL

The carbon arc method was selected as the basis of our microgravity apparatus because it is simple and provides for a fairly large nanotube production rate. However, several challenges were presented in moving the carbon arc from the production lab to the drop tower. The typical carbon arc apparatus<sup>5, 11</sup> employs large 6-12 mm diameter carbon electrodes and consumes multiple kilowatts of power that is supplied continuously from a large welding power supply. The metal catalyst required to produce SWNTs is introduced by drilling holes in the large electrodes and packing them with metal powder. During operation, the arc gap (which determines the current) is usually adjusted manually while observing the arc through a view port. Substantial innovation was required to adapt this configuration to a drop tower rig while maintaining a useful production rate equivalent to or higher than the ground-based machines.

A schematic of the miniature arc apparatus we developed is shown in Figure 2. The carbon electrodes were downsized to 5 mm and new liquid impregnation method was developed to dope the entire rod with  $Ni/Y$  metal catalyst. The arc gap is now continuously adjusted with a spring-loaded electrode. Power for the arc is supplied by a small battery pack that supplies 64 volts and currents in excess of 300 amps. Before producing nanotubes, the reaction chamber is purged of



**Figure 2.** Miniature microgravity carbon arc reactor (chamber dimensions ~20 cm diameter).

air by use of a vacuum. The chamber is then filled with Argon at 600 Torr. The arc vaporizes ~500 mg of carbon during each 2.2 second run, and the resulting SWNTs agglomerate into web like fibers that are easily observed and collected for analysis.

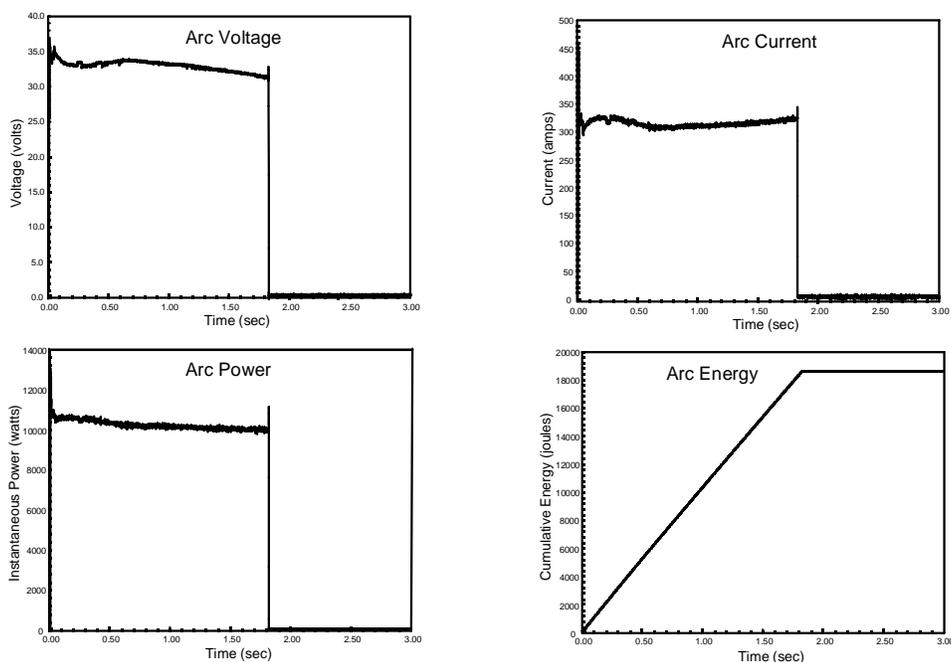
To characterize the performance of the miniature arc, measurements were made of the key electrical parameters such as the arc voltage and current, which determines the carbon vaporization rate and temperature around the arc. Measurements of the voltage and current as well as the instantaneous power and energy deposition for a normal gravity 2-second run are shown on Figure 3. With an arc current of over 300 amps and power of 10kW, this small machine easily outperforms most larger ground based reactors. In addition, as shown by the power and energy curves, the battery pack provides a very stable power source for the duration of the arc, an important factor for providing a uniform nanotube growth environment. When combined with microgravity in the drop tower, this apparatus should provide an ideal environment for studying the formation and growth of carbon nanotubes.

### Summary

Although nanotube science has become one of the worlds most rapidly advancing areas of research, very little is known about the processes involved in nanotube synthesis. To study the formation of carbon nanotubes in an environment unhindered by the buoyancy induced flows generated by the high temperatures necessary to vaporize carbon and grow nanotubes, we have designed a miniature carbon arc apparatus that can produce carbon nanotubes under microgravity conditions. Tests in the 2.2 second and 5.18 second drop towers are planned in 2003. We believe that microgravity processing will allow us to better understand the nanotube formation process and eventually allow us to grow nanotubes that are superior to ground-based production.

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**Figure 3.** Key electrical parameters for the miniature carbon arc.

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